# ENZYME-FILTRATION CLARIFICATION OF XANTHAN GUM POLYMER SOLUTION

### BACKGROUND OF THE INVENTION

This invention relates to removing solids from aqueous solutions of xanthan gum polymers which contain bacterial cell bodies. It is particularly useful for clarifying such polymer solutions for use as water thickeners in aqueous fluids which are injected into subterranean 10 filtering-out of the silica solids and adsorbed cell bodies reservoirs to displace oil.

Numerous procedures have been proposed for clarifying xanthan gum polymer solutions. U.S. Pat. No. 3,966,618 describes treating such solutions with protease enzymes which disintegrate the bacterial cellular 15 debris into water-soluble compounds to an extent such that the polymer solution is clarified. U.S. Pat. No. 3,711,462 describes earlier treatments of such solutions by adding clay particles that are subsequently coagulated and filtered-out so that the cellular bodies are 20 removed along with the coagulated clay particles. U.S. Pat. No. 3,729,460 describes reacting such a debris-containing polymer solution with alkaline materials, at a pH of from about 11.8 to 12.8, to effect a clarification of the

The substantially complete enzymatic disintegration of the cellular bodies tends to convert them to proteinaceous materials which, because they are too finely divided to be removed by filtration, remain in the polymer solution and provide nutrients for bacteria capable 30 of destroying the polymer. The prior non-enzymatic clarification procedures, such as adsorption on coagulated clay particles, or an alkaline treatment prior to a filtration step, tend to be too costly or too difficult for use in waterflooding an oil reservoir.

## SUMMARY OF THE INVENTION

The invention relates to improving a xanthan gum clarification process of the type in which a solution containing the biopolymer and bacterial cell bodies 40 resulting from the fermentation, is treated by disintegrating the cell bodies with a protease enzyme and/or filtering the solution through a finely-bedded filter. The improvement comprises initiating an enzymatic disintegration of the bacterial cell bodies but, at a time at 45 which most of the cell bodies have disintegrated only enough to separate them from the polymer, terminating the cell-disintegrating reaction by (a) contacting the solution with siliceous solids, which have surface areas lent to those of a fine sand, at a pH of from about 10-11, which is capable of causing the adsorption of the partially disintegrated cell bodies on the siliceous solids, and (b) filtering-out the siliceous solids and adsorbed 80% of the bacterial cell bodies are removed from the polymer solution.

# DESCRIPTION OF THE DRAWING

ume with time of the filtrate obtained during the filtration of various aqueous solutions of xanthan gums.

## DESCRIPTION OF THE INVENTION

The present invention is, at least in part, premised on 65 the following. The main benefits of a substantially complete enzymatic disintegration of the bacterial cell bodies and/or an enzymatic or alkaline aqueous solution

treatment of the bacterial cell bodies coupled with filtration through a fine-pore filter, can be attained while also significantly reducing the time, expense and bacteria-nutrient-retaining disadvantages of such treatments. This is accomplished by limiting the extent of the enzymatic disintegration of the cell bodies, by contacting the solution with relatively coarse silica solids onto which the partially disintegrated cell bodies are adsorbed, at a pH that enhances the adsorption, and the through a relatively coarse filter. The present procedure provides a relatively rapid and trouble-free filtration. It also avoids the converting of the cell bodies to unfilterably fine proteinaceous material that can serve as a bacterial nutrient. And, it also avoids the problems associated with micro gel formation within the polymer which have been treated with strongly alkaline solutions.

### ENZYMATIC HYDROLYSIS OR DISINTEGRATION OF BACTERIAL CELL **BODIES**

Novo/Alcalase proteolytic enzyme is known to be useful for use in a biopolymer solution clarification 25 process. It is generally preferable to add the enzyme to an aqueous solution into which the polymer is to be dissolved, prior to or along with, the addition of the polymer. Suitable concentrations of the polymer in the aqueous liquid range from about 300 to 8,000 parts per million (parts by weight). Suitable enzyme treating temperatures range from about 30° to 70° C. (86° to 185° F.). As known to those skilled in the art, the severity or completeness of the treatment increases with both time and temperature, and thus the field conditions would 35 generally dictate how the treating times and temperatures should be adjusted for a particular situation.

The enzyme is effective at relatively low concentration. A 6,000 parts per million Kelzan M.F. polymer concentrate can be clarified with 100 parts per million Novo/Alkalase P 1.5 (available from Novo Enzyme Corporation by a substantially complete disintegration of the bacterial cell bodies, within about 45 minutes at 50° C. In such a solution at such a temperature the partial disintegration contemplated by the present invention would be accomplished in about 10 minutes. During the enzyme treatment, the pH of the aqueous solution is preferably from about 7 to 11. In a particular preferred procedure the pH of a relatively soft aqueous liquid (containing less than about 100 parts per million and adsorptive properties at least substantially equiva- 50 multivalent ion, in terms of calcium ion equivalent) is preferably buffered with a suitable sodium carbonatebicarbonate system, or where the use of hard water is desired such a system containing a chelating agent such as ethylenediamine tetraacetic acid (EDTA), Diquest cell body materials to an extent such that at least about 55 2006 (a salt of an amino tris(methylphosphonic acid) available from Monsanto Chemical Company.

#### PREFERRED ENZYME TREATMENT

The following procedures are outlined in terms of a The drawing shows a graph of the cumulative vol- 60 laboratory procedure but the principles are generally applicable for field use. Additives, such as an oxygen scavenger, an antioxidant, a buffer for a pH of about 10 to 11, and a bactericidal agent, and the enzyme are added to a good quality relatively soft water, or preferably a soft brine containing from about 50 to 5,000 ppm total dissolved salt. The solution is vigorously stirred, or preferably, is sheared, since the enzyme activity is not lost by a relatively high-shear stirring. In a shearing